MOISTURE IN POTATO STARCH*

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Although numerous studies on moisture in cereals and feeds have been conducted, they have not provided a fully satisfactory method for making this determination, and the results are widely divergent. The relatively few data available on moisture in potato starch are especially inconclusive.

An investigation was made of most of the better known methods for the estimation of moisture in agricultural products. The procedures were evaluated, and certain of the methods were modified in order to adapt them to the determination of the moisture content of potato starch. These studies included distillation procedures, the Karl Fischer chemical method, and such oven-drying methods as gravity convection, mechanical convection, and vacuum.

Drying by heat is the procedure commonly used for the determination of moisture in starch. The official method of the German starch trade (14) specifies that five grams of starch be dried in a shallow dish for 1 hour at 50°C. and then for 3 hours at 120°C. The first drying period is intended to

prevent gelatinization of the starch; unless the moisture content is high, however, gelatinization will not take place. Schmorl (10) pointed out that in grains (malt) dried above 105°C. there is a noticeable decomposition of organic material, and it is his opinion that this organic material is essentially starch.

Nelson and Hulett (7) postulate that decomposition of cereals heated in a vacuum occurs at widely different rates at all temperatures, but that until a definite temperature is exceeded the loss from decomposition is slight. They assume that at this temperature such loss becomes measurable and increasingly greater. When plotted, the point where this loss begins is indicated by a break in the moisture-temperature curve. This point does not necessarily show the total amount of moisture present in the substance, but it does show the temperature to which the substance can be safely heated. Then, by extrapolating the first portion of the curve to the critical temperature of water, the total moisture content may be estimated.

Sprockhoff (14) claimed an accuracy of 0.2–0.4 per cent for a method in which 10 grams of starch is supported in a pan suspended by a wire passing through the top of an oven and attached to one arm of a balance beam. The oven is kept at 90°–100°C. for 15–20 minutes and then at 140°–150°C. for 25–35 minutes. The Brabender moisture tester used in some of the drying studies reported in this paper is a modification of this device.

Keller and Keller (4) state that it is possible to dry grain and flour at 160°C. for 10 minutes or at 150°C. for 20 minutes in a Trinkler oven, or at 160°C. for 21 minutes or at 150°C. for 25 minutes if an air oven is used. Eva, Milton, and Geddes (2) show that the 130°C. air-oven, the gravity convection Brabender oven, and the DeKhotinsky oven give values for moisture that are similar to the vacuum oven (about 0.1 per cent lower).

The oven methods employed by Sair and Fetzer (8, 9) in their investigation of moisture determination in corn yielded results that ranged from a low value for the air oven at 110°C. to a high value for the vacuum oven at 100°C. They demonstrated that corn dried in the air oven still contained moisture by the fact that use of P_2O_5 in conjunction with the air oven gave a value comparable to that obtained with the vacuum oven.

The official method of the English starch trade specifies the use of the Carter-Simon moisture tester, which follows the principle of higher temperature for short drying periods. The oven is equipped with a chimney to give a more rapid circulation of air in the drying compartment, which is relatively small in proportion to the size of the drying dishes and to the chimney displacement. A 5 gram sample is dried for 15–30 minutes at 155°C.

Snyder and Sullivan (11, 12, 13) reported that when flour is dried dextrinization takes place, followed by a marked change in the solubility of the carbohydrates, in the color of the flour, and in other physical and

chemical characteristics. The dextrinization of the starch, together with dehydration of the proteins during drying, and elimination of water in both cases, account for the anomalous moisture results commonly observed in the determination of moisture in flour.

Distillation procedures for moisture determination in which toluene, xylene, light petroleum, and other solvents lighter than and immiscible with water are used have been found satisfactory for some materials. Sair and Fetzer (8, 9) found that distillation with toluene and with benzene yielded results that were similar and that compared favorably with their established moisture content. Alexander (1) reported the use of carbon tetrachloride, a liquid heavier than water, with a special trap so designed that the column of carbon tetrachloride would support the condensed water column. This has certain advantages over the lighter liquids in that it is nonflammable and keeps the sample from lying on the bottom of the flask, thus preventing superheating and charring.

It may require hours to distil over all the water with the lower-boiling liquids, while the use of high-boiling liquids leads to decomposition of the material with the production of water. Tausz and Rumm (15) and Fairbrother and Wood (3) describe methods of distillation with tetrachloroethane, another solvent that is heavier than water and has a higher boiling point than xylene or toluene, but they do not consider the possible decomposition of the carbohydrate material. Moreover, they use as their standard of comparison gravity convection-oven drying at 98.5°C. This is an empirical procedure, as it measures the water liberated at that temperature, but does not necessarily measure the total moisture content of the sample.

Tucker and Burke (16) report that distillation of most cereal products with tetrachloroethylene gives values that are the same in a few cases but usually are higher than those obtained by drying at 98.5°C. Lampe (5), who investigated starchy materials, reports that his results show the tetrachloroethylene procedure to be suitable for routine moisture determinations.

In the investigation of the use of the Karl Fischer reagent for the chemical determination of moisture in regenerated cellulose, paper, and wood, Mitchell (6) reports that the method gives a more nearly complete recovery of moisture than do the oven-drying methods. He obtained consistently higher results with the Karl Fischer reagent than by drying the same material at 102°C. to constant weight. It does not necessarily follow, however, that these results represent the actual moisture content, since the oven-drying procedure at 102°C. may not have removed all the moisture.

METHODS USED

Three samples of potato starch and one of rice starch were obtained. Each sample was thoroughly mixed by rolling it on a sampling cloth and

then stored in moisture-tight Mason jars. The following discussion is mainly concerned with the results obtained with Sample 1, potato starch.

Mechanical Convection-Oven Methods.—Two types of mechanical convection ovens were used, a Brabender moisture tester and a Precision floor model.

The Brabender moisture tester, an air oven with forced circulation, has a balance incorporated in the apparatus in such a manner that the sample can be weighed without removing it from the drying chamber. As the loss in weight is read directly in per cent on an illuminated scale, it can be measured at any interval of time for a given temperature, thus eliminating errors inherent in methods that require removal of the sample from the oven, cooling before weighing, and reheating to constant weight. Thus the rate of loss in weight caused by heat at any given temperature can be followed closely. Duplicate 5 gram (±0.005) samples were weighed into tared, shallow aluminum pans (85 mm. ×15 mm.) and placed in the drying chamber, which had been regulated to a given temperature. The loss in weight was noted at definite time intervals until a constant reading was obtained. This procedure was repeated for each temperature under consideration. All samples used in this study were weighed initially at the same time and stored in a desiccator containing no desiccant.

In addition to the Brabender, a Precision floor-model mechanical convection oven and tared aluminum moisture dishes (50 mm. ×25 mm.) with tight-fitting covers were used. Tests were made, either in duplicate or in triplicate, on 1–5 gram samples. The dishes were uncovered during the drying period. All the samples for each drying temperature were weighed and placed in the oven simultaneously, and each sample was removed after a definite time interval, cooled, and weighed. Since by this procedure the time required to reach constant weight at the temperature under consideration was determined without repeated periods of heating and cooling, the only opportunity for error was in the single cooling process.

Gravity Convection-Oven Methods.—A Precision table-model gravity convection oven was used for many of the air-oven tests. The same moisture dishes and weights of sample were used as were employed in the mechanical convection oven, and the method of obtaining the degree of drying was the same as with the Precision mechanical convection oven.

The Carter-Simon tests were made on 5 gram (±0.005) samples, which were weighed out in the shallow, open, Carter-Simon moisture pans at the same time. Each sample was run through the oven at a different rate. Thus the time that each sample was in the drying chamber was varied. This procedure was repeated for each temperature of the experiment.

Vacuum-Oven Drying Method.—A Weber vacuum oven was used. The pressure in the drying chamber was maintained at less than 5 mm. of mercury by use of a Welch "Duo-seal" pump. The same dishes, weight of

sample, and method of controlling errors were used as for the precision mechanical convection-oven drying method.

Distillation Methods.—The apparatus used for solvents heavier than water was the Hercules moisture tester (1); for solvents lighter than water, a modified Bidwell-Sterling apparatus was used. Twenty-gram samples were weighed into the distillation flasks, and sufficient solvent (100 ml.) was added to fill the receiver and cover the sample. The receivers had a capacity of 5 ml. graduated in 0.05 ml. Distillation was continued until the volume of the trapped water remained constant (3–5 hours).

Chemical Method.—The Karl Fischer chemical method for the determination of moisture was used. The reagent was prepared according to the Mitchell (6) modification of the original Karl Fischer formula. One-gram samples were extracted with 10 ml. of water-free methanol for 8 hours in 125 ml. Erlenmeyer flasks covered with a piece of rubber dental dam. The titration was carried out by inserting the buret tip through a pinhole in the rubber, thus preventing moisture in the laboratory air from coming in contact with either the reagent or the reaction mixture, thus making possible a sharp end point and reproducible results.

DISCUSSION

The percentage loss in weight of potato starch recorded by the Brabender moisture tester becomes greater with successively higher temperatures (Table 1). The rate of loss at each temperature is high in the beginning and falls off rapidly until the weight of the sample remains constant. In Figure 1 the percentage loss in weight is plotted against time. The curve for 120°C. lies just above that for 100°C., and the curve for each temperature is higher than the one for the temperature immediately below it. These curves show that the total amount of volatile material liberated is dependent upon the temperature used. At the higher temperatures a light-brown discoloration appears, indicating an alteration in some of the samples during the time necessary to establish the constancy of weight. This alteration, however, does not affect the weight of the sample, as was demonstrated by a test run at a temperature above that for any run illustrated in Figure 1. Table 2 reports the data on potato starch dried in the mechanical convection oven for varying lengths of time at 180°C. The spectral reflectance of each of the samples was measured with a General Electric recording spectrophotometer, freshly deposited magnesium oxide being used as the standard. Parallel iodine tests were made on water extracts of the samples to indicate changes in composition and solubility. In Figure 2 values of −log R, where R equals reflectance at 436 millimicrons, are plotted against log T (time in minutes). Increasing values of −log R correspond with increasing intensity of color as viewed by the eye. From Figure 2 and Table 2 it may be observed that no noticeable change

TABLE 1.—*Loss in weight of potato starch[a] dried in Brabender moisture tester at various temperatures for different periods of time*

100°C.		120°C.		135°C.		150°C.[b]		155°C.[b]		160°C.		170°C.	
TIME	LOSS IN WT.	TIME	LOSS IN WT.	TIME	LOSS IN WT.	TIME	LOSS IN WT.	TIME	LOSS IN WT.	TIME	LOSS IN WT.	TIME	LOSS IN WT.
min.	*per cent*	*min.*	*per cent*	*min.*	*per cent*	*min.*	*per cent*	*min.*	*per cent*	*min.*	*per cent*	*min.*	*per cent*
2	3.15	2	3.40	2	4.40	1	5.20	1	9.20	1	2.25	1	2.40
5	6.25	4	7.40	4	9.05	2	7.50	2	10.95	2	5.85	2	7.20
9	10.20	6	10.30	6	12.00	3	10.85	3	11.50	3	8.90	3	10.80
12	11.95	8	12.20	7	13.00	4	11.70	4	13.80	4	11.20	4	13.25
15	13.00	10	13.30	8	13.80	5	12.85	5	14.60	5	12.90	5	14.80
18	13.85	12	14.20	9	14.35	6	13.90	6	15.20	6	14.10	6	15.90
21	14.35	14	14.80	10	14.85	7	14.60	7	15.60	7	15.10	7	16.40
24	14.80	16	15.35	11	15.30	8	15.30	8	16.00	8	15.70	8	16.50
27	15.25	18	15.60	12	15.50	9	15.70	9	16.25	9	16.15	9	16.75
30	15.30	20	15.70	13	15.75	10	16.15	10	16.35	10	16.35	10	16.80
33	15.50	21	15.80	14	15.90	11	16.25	11	16.50	11	16.55	11	16.80
36	15.55	22	16.05	15	16.00	12	16.45	12	16.60	12	16.65	12	16.85
39	15.60	24	16.05	16	16.20	13	16.45	13	16.60	13	16.70	13	16.85
42	15.85	26	16.10	17	16.30	14	16.45	14	16.60	14	16.70	14	16.85
45	15.85	28	16.15	18	16.30	15	16.45	15	16.65	15	16.70	15	16.90
48	15.90	30	16.20	19	16.30	16	16.50	16	16.65	17	16.80	16	16.85
51	15.95	32	16.20	20	16.30	17	16.55	17	16.65	19	16.80	17	16.85
54	15.95	34	16.20	21	16.30	18	16.60	18	16.70	22	16.85	20	16.90
57	16.00	36	16.35	22	16.35	19	16.60	19	16.70	25	16.85	25	16.90
60	16.00	38	16.40	23	16.35	21	16.60	20	16.70	30	16.85	30	16.90
63	16.00	40	16.35	25	16.40	24	16.60	22	16.70	42	16.85	40	16.90
68	16.00	42	16.30	27	16.35	26	16.60	25	16.70	52	16.85	50	16.90
74	16.00	45	16.40	29	16.35	29	16.55	30	16.70	60	16.85	60	16.90
95	16.00	50	16.35	31	16.40	30	16.55	40	16.70				
122	16.00	60	16.30	33	16.40	34	16.60	50	16.70				
164	16.00			35	16.40	39	16.60	60	16.70				
				39	16.35	44	16.65	70	16.70				
				44	16.40	50	16.65	80	16.70				
				50	16.35	60	16.55						
				60	16.40								

[a] Sample 1.
[b] Sample was stored in empty desiccator for 2 days before the determination was made, but was weighed at the same time as all other samples.

in the reflectance of the starch and no change in solubility occurred until the constant-weight period was reached. During the constant-weight period, a gradual increase in the discoloration ($-\log R$) was noted, paralleled by a gradual increase in the solubility of the starch, as indicated by the iodine reaction. Although discoloration and solubilization were proceeding during the plateau period, they did not affect the weight of the sample, as indicated by the constancy of weight over a period of nearly four hours. After four hours of heating, a more drastic breakdown or alteration occurred, accompanied by a further loss in weight, a sharp rise in the rate of discoloration, and the production of erythrodextrins, as was shown by the production of red color in the iodine test. Thus loss in weight occurred only after much longer time intervals than were necessary to establish the constant weight value. Since this was true when drying at 180°, it must be true likewise for drying at lower temperatures, as illus-
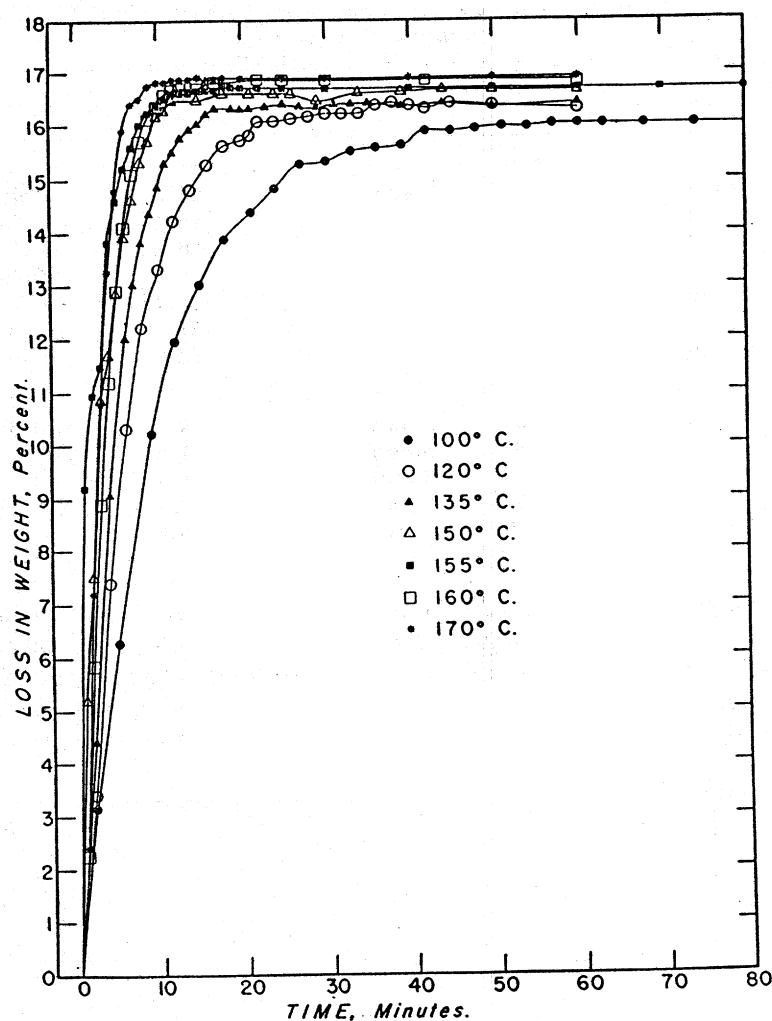
FIG. 1.—LOSS IN WEIGHT OF POTATO STARCH (SAMPLE 1) DRIED IN THE BRABENDER MOISTURE TESTER AT VARIOUS TEMPERATURES FOR DIFFERENT PERIODS OF TIME.

trated graphically in Figure 1. These data also confirm Nelson and Hulett's observations (7) that decomposition occurs at widely different rates and that until a definite time is reached the loss from decomposition is slight, even at a high temperature, but that after this point the loss from decomposition becomes increasingly greater.

If the loss that occurs in reaching constant weight is plotted against the respective temperature for each isotherm of Figure 1, a moisture-temperature curve such as that shown in Figure 3 results. The low temperature end of this curve is rather steep, the loss in weight being sensitive to slight dif-

TABLE 2.—*Effect of drying potato starch (Sample 1) at 180°C. in mechanical convection oven*

TIME	LOSS IN WT.	COLOR	REFLECTANCE AT 436 Mμ	IODINE TEST
minutes	*per cent*		*per cent*	
Control	—	White	90.3	−Colorless
1	0.78	White	90.2	−Colorless
4	7.08	White	90.2	−Colorless
8	13.57	White	89.9	−Colorless
12	16.17	White	89.4	−Colorless
20	16.67	Very light brown	87.4	+Faint green
30	16.67	Light brown	80.0	+Green
Hours				
1	16.69	Brown	69.5	++Blue
2	16.66	Brown	61.2	++Blue
4	16.71	Brown	51.4	++Purple
24	17.51	Dark brown	9.7	+++Red
48	18.89	Dark brown	5.8	++++Red

ferences in temperature. As the temperature increases, the curve flattens out, the loss in weight apparently becoming less sensitive to temperature variations. Then above a certain temperature, the curve again becomes steep, and smaller changes in temperature produce greater changes in weight. Thus, the curve is divided into two distinct portions, which can
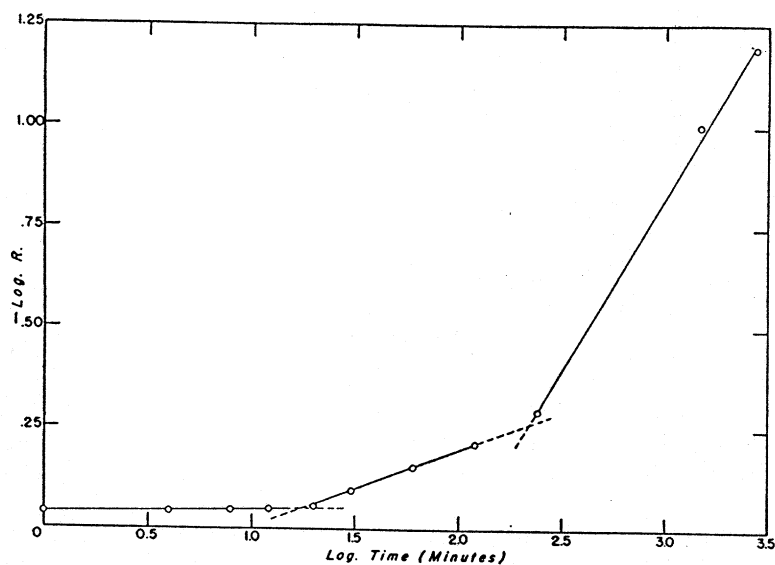

FIG. 2.—REFLECTANCE OF POTATO STARCH (SAMPLE 1) DRIED AT 180°C. IN A MECHANICAL CONVECTION OVEN FOR VARIOUS PERIODS OF TIME.

be extended until they intersect, the intersection being apparently the maximum temperature for the flat portion of the curve.

Any method for the determination of moisture based on the steep portion of the curves of Figure 1 or the upper portion of the curve of Figure 3 is subject to considerable error produced by relatively small changes in time (Figure 1) or in temperature (Figure 3). This may account for the inconclusive data reported in the literature for moisture content of potato starch, since most of the standard methods specify periods of time or temperatures that fall on the steep portions of the respective curves. Therefore, a more nearly reproducible method for the determination of
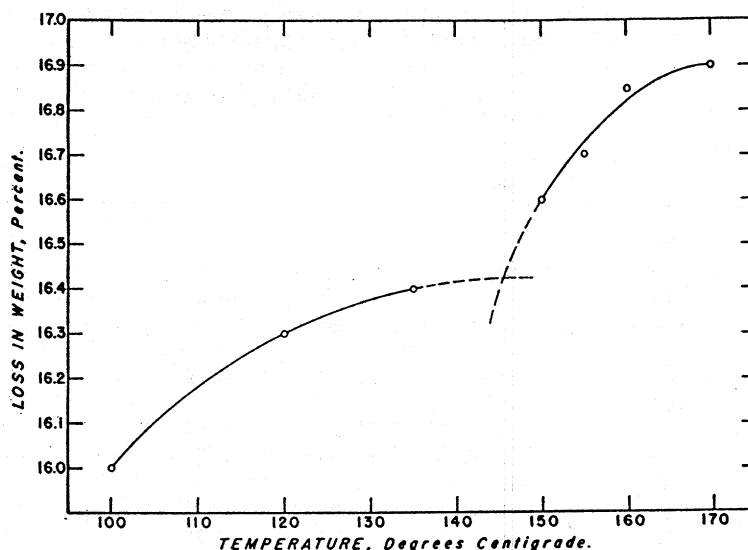
FIG. 3—LOSS IN WEIGHT OF POTATO STARCH (SAMPLE 1) DRIED AT VARIOUS TEMPERATURES IN BRABENDER MOISTURE TESTER.

moisture in potato starch would consist of the use of a temperature indicated on the flattest portion of the moisture-temperature curve and of a time sufficiently long to insure that constant weight has been reached. With the Brabender moisture tester the method would consist of heating at a temperature of 135°–145°C. for 30–60 minutes or to constant weight. For Sample 1 this would give a moisture content of 16.43 per cent.

Nelson and Hulett (7) suggested that the first portion of a similar curve be extrapolated to the critical temperature of water to obtain the true moisture content of the sample. When their data were plotted, an apparent straight line was obtained, which was interpreted to indicate that the increase in loss of moisture was directly proportional to the increase in drying temperature. A critical study of their data, however, reveals that the slope at each point on the curve is not a constant, which excludes the pos-

sibility of a straight line. This is further proved by replotting their data on a larger scale, which results in a curve very similar to that in Figure 3. The extrapolation of a curve requires many more points and a much higher degree of accuracy than the extrapolation of a straight line. Since these conditions are experimentally unattainable under the present circumstances, it does not seem possible to make this extrapolation. Furthermore, the flattening of the curve before the break suggests that little or no moisture remains in the sample. Hence, there seems to be no need to extrapolate, and the percentage indicated by the break has been taken as the moisture content.

The cause of the greater loss in weight at temperatures higher than the

TABLE 3.—*Loss in weight of starch heated to constant weight at various temperatures in Brabender moisture tester*

	LOSS IN WEIGHT (%)			
TEMPERATURE	POTATO STARCH			RICE STARCH
	SAMPLE 1	SAMPLE 2	SAMPLE 3	
°C.				
90				7.95
100	16.00			8.25
110		10.95	6.25	8.70
120	16.30	11.35	6.35	9.55
130				9.55
135	16.40	11.40	6.45	
140				9.60
145		11.45	6.55	
150	16.60	11.50	6.65	9.75
155	16.70			
160	16.85	11.75	6.90	9.95
170	16.90	11.90	7.10	10.10

break is undetermined, but it may be decomposition or a more deep-seated dehydration of the starch molecule. Since the slope of the curve just above the break is steep and the curve below the break is nearly flat, it may be considered that the extent of decomposition or deep-seated dehydration at temperatures up to the break is negligible.

That the temperature at which the break in the moisture-temperature curve occurs is unaffected by the initial moisture content was confirmed with two other samples of potato starch obtained from different sources and having different moisture contents. The results show that for potato starch the point at which the break occurs can be duplicated at 145°C. (±2°). These data are reported in Table 3 and illustrated graphically in Figures 4 and 5. As a check, a new aliquot of each of Samples 2 and 3 was dried to constant weight in the Brabender moisture tester at 145°C. The cross on Figures 4 and 5 represents the percentage loss for each aliquot.
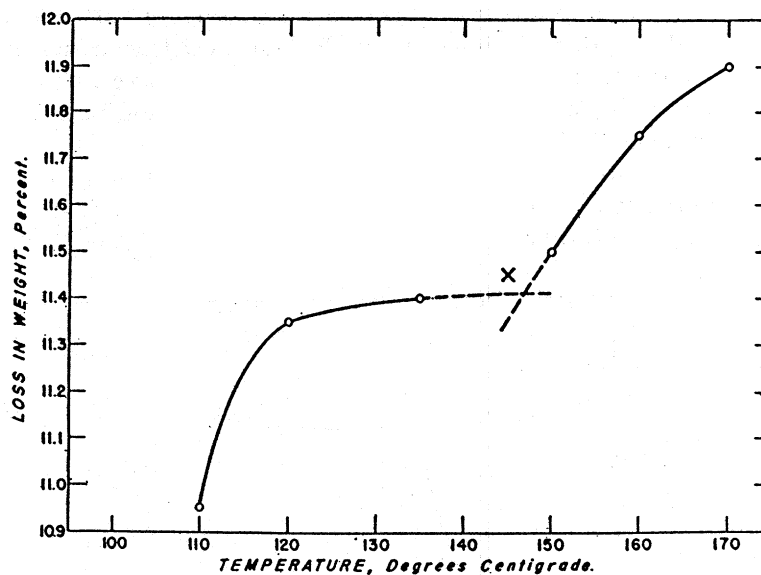
FIG. 4.—LOSS IN WEIGHT OF POTATO STARCH (SAMPLE 2) DRIED AT VARIOUS
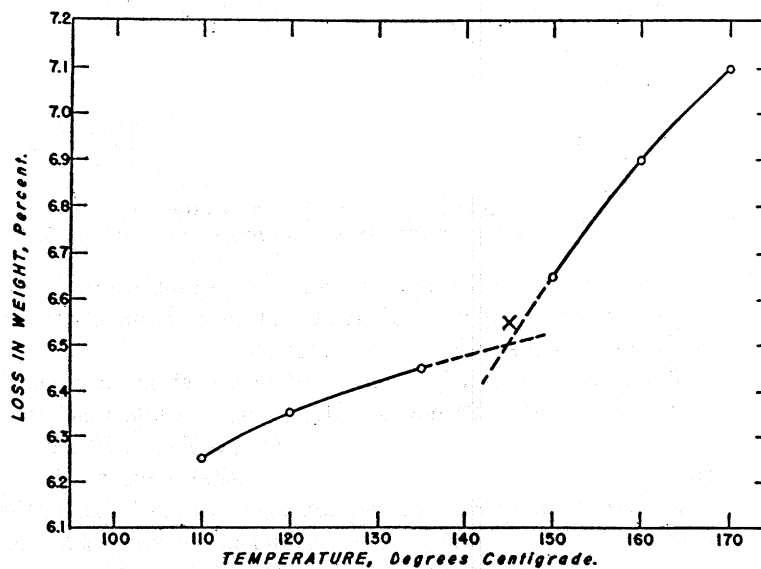TEMPERATURES IN BRABENDER MOISTURE TESTER.

FIG. 5.—LOSS IN WEIGHT OF POTATO STARCH (SAMPLE 3) DRIED AT VARIOUS
TEMPERATURES IN BRABENDER MOISTURE TESTER.

These values check the loss in weight at the break within the reading accuracy of the instrument (± 0.05 percent). If the type of starch is different, however, a new temperature range will be found. For example, a curve for

rice starch* results in a flat portion with a range of 120°–135°C. (Figure 6).

Hence, there is proposed a method for the determination of moisture in potato starch that consists in drying the sample to constant weight in the Brabender moisture tester or some similar apparatus at a temperature range of 135°–145°C.

Now that a basic method has been found that gives reproducible results in the determination of moisture in potato starch, it should be possible to employ any other method that will duplicate these moisture values within
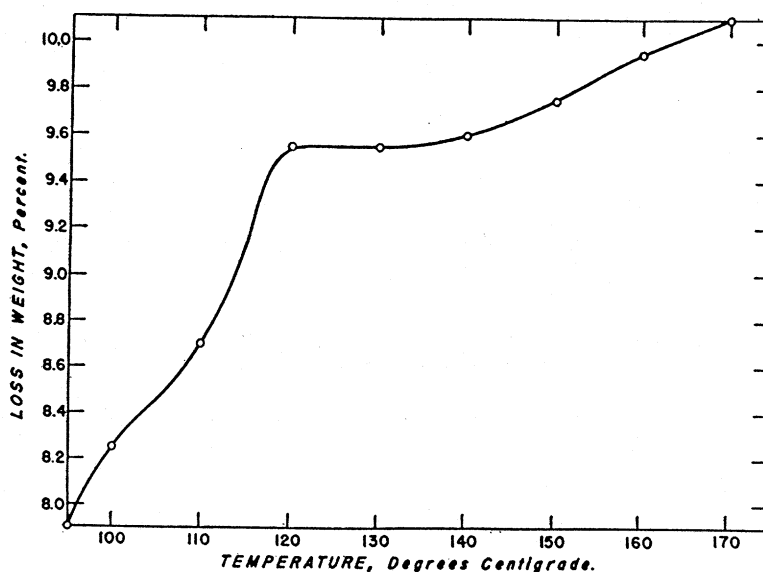
FIG. 6.—LOSS IN WEIGHT OF RICE STARCH DRIED AT VARIOUS
TEMPERATURES IN BRABENDER MOISTURE TESTER.

the limit of error of the specific procedure in question. Therefore, the following experiments were planned and carried out to find which of the common methods fulfill these conditions.

When starch was dried in a gravity-convection air oven at 100°C., the loss in weight after 24 hours of drying never quite reached the value obtained in the mechanical convection air oven at 100°C. Furthermore, the rate of drying in the mechanical convection oven was much more rapid than that in the gravity convection oven (Table 4). In neither case, however, did the value reach that established by the basic procedure mentioned above (see Figure 3). Table 4 also shows that vacuum-oven drying of starch under a pressure of approximately 5 mm. of mercury at 80°C. resulted in a moisture value in close agreement with that obtained by the basic method and that constant weights were reached after 22–24 hours.

TABLE 4.—*Loss in weight of potato starch (Sample 1) dried at various temperatures in ovens of different types*

TEMPERATURE	AIR OVEN		MECHANICAL CONVECTION OVEN		VACUUM OVEN (PRESSURE CA. 5 MM.)		CARTER-SIMON OVEN	
	TIME	LOSS IN WT.	TIME	LOSS IN WT.	TIME	LOSS IN WT.	TIME	LOSS IN WT.
°C.	hours	per cent	hours	per cent	hours	per cent	minutes	per cent
80					1.5	15.48		
					22	16.35		
					24	16.48		
100	1	13.78	1	15.84	1	16.47		
	2	15.20	2	15.98	5	16.56		
	3	15.60	22	15.92	24	16.58		
	24	15.61	67	16.07				
135	2	16.39	1	16.43			15	14.66
	5	16.58	12	16.53			30	16.24
	22	16.58					60	16.24
150	$\frac{1}{2}$	16.66	$\frac{1}{4}$	16.44			10	13.86
	1	16.66	$\frac{1}{2}$	16.60			15	16.47
	2	16.65	1	16.71			20	16.52
	3	16.77	3	16.65			30	16.52
180			minutes					
			1	0.78				
			4	7.08				
			8	13.57				
			12	16.17				
			20	16.67				
			hours					
			$\frac{1}{2}$	16.67				
			1	16.69				
			2	16.66				
			4	16.71				
			24	17.51				
			48	18.89				

Vacuum drying at 100°C. gave a slightly higher value, but the weights were constant after 5 hours. Drying at 100°C. by any of the other methods gave lower values. At 135°C. the air-drying methods used, with the exception of the Carter-Simon, gave results comparable with those of the proposed method.

On heating the starch to constant weight at higher temperatures (150°C. and above), the loss in weight was greater than that caused by loss of moisture. Quick approximations of the moisture content can be made, however, by heating at one of these high temperatures for a length of time that will give a loss in weight comparable to the moisture content estab-

TABLE 5.—*Moisture in potato starch (Sample 1) determined by distillation with various immiscible solvents*

SOLVENT	BOILING POINT	H_2O
	°C.	per cent
Carbon tetrachloride	76	15.50
Toluene	110–111	16.20
Xylene	137–140	16.33
Tetrachloroethane	146	16.40

lished by the basic method. Heating must be stopped at the expiration of this specific time, since the time selected would fall on the steep portion of a curve similar to those in Figure 1. For example, when samples were heated at 150°C. in the mechanical-convection air oven for 15 minutes, values approximating the accepted ones for moisture were obtained. This principle is also utilized by the Carter-Simon moisture tester, which is operated at 155°C. for 15 minutes (the time and temperature recommended by the manufacturer for drying starch). When the same time and temperature were used with the Carter-Simon moisture tester the results agreed with the moisture content established by the basic method (Table 4). It must be kept in mind, however, that rapid drying at higher temperatures gives only approximate values.

TABLE 6.—*Summary of loss in weight (per cent) of potato starch (Sample 1) dried to constant weight at various temperatures[a] by different methods*

TEMPERATURE	TYPE OF OVEN					
	BRABENDER LOSS IN WT.	MECHANICAL CONVECTION LOSS IN WT.	CARTER-SIMON LOSS IN WT.	AIR LOSS IN WT.	DISTILLATION LOSS IN WT.	VACUUM LOSS IN WT.
°C.						
76						
80					15.50	
100	16.00	16.00				16.48
110				15.61		16.57
120	16.30				16.20	
135	16.40	16.53	16.24			
137				16.58		
146					16.33	
150	16.55	16.65			16.40	
155	16.70			16.66		
160	16.85		16.52			
170	16.95					
180		16.68				

[a] Areas enclosed indicate the temperature range that may be used for determining the moisture content of potato starch by any given drying method.

Distillation methods for the determination of moisture gave results of the same order as those given by the oven-drying methods (Table 5). Generally, as the temperature of distillation was raised, the volume of water distilled from the sample increased. Because of their low boiling points, 76° and about 110°C., carbon tetrachloride and toluene gave results that were too low, as indicated by the steep part of the lower portion of the curve in Figure 3. Xylene and tetrachloroethane, whose boiling points would fall on the flat portion of the curve in Figure 3, gave percentages of moisture in agreement with those of the basic method.

TABLE 7.—*Moisture in potato starch (Sample 1) determined by Karl Fischer reagent*

RUN NO.	H_2O
	per cent
1	16.14
2	16.01
3	16.17
4	16.04
5	16.00
6	16.08
7	16.00
Average	16.06

Owing to the low specific gravity of xylene, starch settles out when this solvent is used, and care should be taken to prevent charring on the bottom of the flask. In this experiment charring was avoided by slow distillation and intermittent shaking of the distilling flask. With tetrachloroethane, the distillation could be carried out more rapidly, since the starch floated on the surface of the liquid.

Table 6 gives a summary of the percentage losses in weight of potato starch dried to constant weight by different methods of various temperatures. The moisture values by the basic method and close approximations to it by other methods are indicated by enclosing lines.

The percentage of moisture in Sample 1, potato starch, determined by the Karl Fischer reagent is 16.06 per cent (Table 7), which is considerably lower than that obtained by the basic method (16.43 per cent), but higher than the values obtained by gravity-convection air oven drying at 100°C. (15.60 per cent).

Most of the data reported in the literature for the determination of moisture by the Karl Fischer reagent were compared with results obtained by drying in gravity-convection air ovens at approximately the boiling point of water (100°C.). They show, almost without exception, that the chemical method gives higher results. The conclusion drawn was that recovery of moisture by the chemical method is more nearly complete than

is recovery by oven-drying methods at or around the temperature of boiling water (6). However, if the results by the chemical method were compared with those obtained by oven drying in the range 135°–145°C., the recovery of moisture would be found to be less complete by the chemical method than by the oven-drying methods.

SUMMARY

The effects of time and temperature on the loss in weight of potato starch by oven drying have been studied. From this study the conditions of drying that give the most nearly reproducible values for moisture content have been determined. By use of a Brabender moisture tester it was established that heating to constant weight at temperatures in the range of 135°–145°C. gives reproducible values. By using this basic procedure, it was found possible to modify several of the commonly used methods and obtain values in agreement with results established by the basic method. These methods have been found to be applicable to the determination of moisture in various agricultural products such as leafy vegetables and certain natural rubber-bearing plants, but experience indicates that the optimum conditions of time and temperature must be determined for each particular material.

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